INTERNATIONAL STANDARD

ISO 15968

Second edition 2016-02-01

Direct reduced iron — Determination of apparent density and water absorption of hot briquetted iron (HBI)

Minerais de fer préréduits — Détermination de la masse volumique apparente et de l'absorption d'eau du fer briqueté à chaud



ISO 15968:2016(E)



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 3, *Physical testing*.

This second edition cancels and replaces the first edition (ISO 15968:2000), which has been technically revised with the following changes:

- to homogenize its structure and wording with other physical test standards;
- to contemplate the outcomes of the studies on mass definition.

Introduction

This test method has been developed to determine the apparent density and water absorption of direct reduced iron in the form of hot briquetted iron (HBI).

Results of this test have to be considered in conjunction with other tests used to evaluate the quality of products from direct reduction processes.

This International Standard can be used to provide test results as part of a production quality control system, as a basis of a contract or as part of a research project.

The apparent density measured in this test can be used to certify that the HBI meets the apparent density requirements of the International Maritime Organization (IMO) Code of Safe Practice for Solid Bulk Cargoes.

Direct reduced iron — Determination of apparent density and water absorption of hot briquetted iron (HBI)

CAUTION — This International Standard may involve hazardous operations and equipment. This International Standard does not purport to address all of the safety issues associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.

1 Scope

This International Standard specifies a method of determining the apparent density and water absorption of direct reduced iron by immersion in water.

This International Standard is applicable to hot briquetted iron (HBI).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3310-1, Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth

ISO 3310-2, Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate

ISO 10835, Direct reduced iron and hot briquetted iron — Sampling and sample preparation

ISO 11323, Iron ore and direct reduced iron — Vocabulary

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

4 Principle

Dried briquettes are weighed in air, soaked in water, surface-dried and weighed again: first in air and then in water. The apparent density and water absorption are determined by water absorption method.

5 Sample, sample preparation and preparation of test portions

5.1 Sampling and sample preparation

Sampling of a lot of HBI and sample preparation shall be in accordance with ISO 10835.

A test sample of a sufficient quantity to provide at least 100 briquettes shall be obtained.

Sieve the test sample by hand on a 40 mm test sieve to discard any -40 mm material.

5.2 Preparation of test portions

Spread the test sample on a smooth and flat plate to form a single layer of briquettes, in the shape of a rectangle.

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At least 4 test portions, each of at least 6 briquettes, shall be prepared by taking at random single briquettes.

Oven-dry the test portion chosen for testing to constant mass at $105\,^{\circ}\text{C} \pm 5\,^{\circ}\text{C}$. Cool the test portion in air to room temperature and remove any adhering dust from the individual briquettes with a soft brush, or by gently blowing with compressed air.

6 Apparatus

6.1 General

The test apparatus shall comprise the following:

- a) ordinary laboratory equipment, such as an oven, hand tools and safety equipment;
- b) two vessels;
- c) suspension device;
- d) test sieves;
- e) weighing device.

<u>Figure 1</u> shows an example of the test apparatus.

6.2 Two vessels, to contain water, one for soaking the briquettes in water and the other for weighing soaked briquettes in water on a top loading balance.

The second vessel should be large enough to ensure that the suspended briquette, or a wire basket holding the briquettes, is completely submerged and does not touch the sides or bottom of the vessel, e.g. a vessel, 200 mm in diameter by 200 mm high, is sufficient for the basket specified in <u>6.4</u>.

- **6.3 Suspension device**, to allow the test pieces to be suspended and weighed in water.
- **6.4 Suspension wire or wire basket**, to support the briquettes when suspended from the suspension device.

NOTE A wire basket, 150 mm in diameter by 100 mm high, is sufficient to hold a test portion of 6 typical briquettes.

- **6.5 Water**, free from any impurity (for example dissolved air) that would significantly affect its density.
- In case of doubt, use distilled or deionized water, or filtered water that has been freshly boiled.
- **6.6 Balance**, top loading, capable of weighing the vessel with the water soaked briquettes and an accuracy of 0,1 g.
- **6.7 Cloth or paper towel**, to surface-dry the test piece.
- **6.8** Thermometer, adequate to measure the temperature of the water used for immersion.
- **6.9 Test sieve**, conforming to ISO 3310-1 or ISO 3310-2 and having square aperture with a nominal size of 40,0 mm.
- **6.10 Weighing device**, capable of weighing the test sample and test portions to an accuracy of 1 g.

7 Procedure

7.1 Number of determinations for the test

Carry out the test as many times as required by the procedure in Annex A.

7.2 Density determination

Take, at random, one of the test portions prepared in <u>5.2</u>. The briquettes in the test portion may either all be tested at the same time or be tested individually in random order and the results averaged.

Determine and record the mass (m_1) of the test portion.

Soak the dried briquettes of the test portion in a vessel $(\underline{6.2})$ containing water at a temperature of 22 °C ± 5 °C. The briquettes may be soaked all at one time or individually. Completely submerge them in the water to soak, and turn or jiggle them occasionally to help remove air bubbles. Allow the briquettes to remain submerged until all air bubbling stops. This may take up to 60 min.

Remove the soaked briquettes from the vessel. Let them drain momentarily and then gently surfacedry them with paper towels (6.7) or a damp towel taking care not to draw water out of any of the pores. Place them on a weighing device (6.10) and immediately weigh. Determine and record the total mass (m_2) of all the soaked briquettes in the test portion.

Determine the mass of soaked briquettes in water. Two methods are allowed for this step.

Method 1 — Testing of entire test portion — Wire basket method

Place a vessel containing water at 22 °C ± 5 °C on a top loading balance and tare to zero. Note that the vessel shall have enough freeboard to accommodate the water that will be displaced by the test portion without overflowing (approximately 600 ml for 6 average-sized briquettes).

Suspend the empty wire basket (6.4) in the water. Record the balance reading (m_3) .

Vertically raise the wire basket out of the water and place each of the previously weighed surface-dried soaked briquettes into the basket, being careful not to splash any water out of the vessel. Lower the basket containing the briquettes back into the water. Ensure that the briquettes are completely submerged and that the cage hangs free and does not touch the sides or bottom of the vessel. There should be no air bubbling and the weight should be constant immediately. Record the balance reading (m_4) .

Method 2 — Testing individual briquettes — Wire suspension method

Place a vessel containing water at 22 °C ± 5 °C on a top loading balance and tare to zero. Suspend individually the previously weighed surface-dried soaked briquettes in the tared vessel. Completely submerge each briquette in the water making sure that it hangs free and does not touch the sides or the bottom of the vessel. There should be no air bubbling and the weight should be constant immediately. Record the balance reading $[m_{5(1)}]$. Repeat the operation for each briquette in the test portion and record the balance reading for each briquette, $[m_{5(2)}, ..., m_{5(6)}]$. Sum the masses of the 6 briquettes and record the result (m_5) .

8 Expression of results

8.1 Calculation of the apparent density (ρ_a)

The apparent density, ρ_a , is calculated from Formula (1) or Formula (2):

a) Wire basket method
$$\rho_a = \frac{m_1 \times \rho_w}{(m_4 - m_3)}$$
 (1)

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b) Wire suspension method
$$\rho_a = \frac{m_1 \times \rho_w}{m_5}$$
 (2)

where

 m_1 is the mass in air, in grams, of the dried briquettes;

 m_3 is the mass, in grams, of the empty wire basket immersed in water;

 m_4 is the mass, in grams, of the soaked briquette plus wire basket immersed in water;

 m_5 is the sum of the masses, in grams, of the six soaked briquette immersed in water;

 ρ_w is the water density, in g/cm³.

Record the results of the density determinations of each test portion (at least 6 briquettes) to 2 decimal places.

8.2 Calculation of the water absorption (a)

The water absorption, *a*, expressed as a percentage of the dry mass, is calculated from Formula (3):

$$a = \frac{(m_2 - m_1)}{m_1} \times 100 \tag{3}$$

where

 m_1 is the mass in air, in grams, of the dried briquettes;

 m_2 is the mass in air, in grams, of the surface-dried soaked briquettes.

Express the result rounded off to the nearest 0,1 %.

8.3 Repeatability and acceptance of test results

Follow the procedure in Annex A by using the repeatability r = 0.2 (g/cm³) for apparent density. Results shall be reported to two decimal places. Repeatability for the water absorption, a, can be neglected.

9 Test report

The test report shall include the following information:

- a) a reference to this International Standard, e.g. ISO 15968;
- b) all details necessary for the identification of the sample;
- c) the name and address of the test laboratory;
- d) the date of the test;
- e) the date of the test report;
- f) the signature of the person responsible for the test;
- the details of any operation and any test conditions not specified in this International Standard or regarded as optional, as well as any incident which may have had an influence on the results;
- h) the apparent density, ρ_a ;
- i) the water absorption, *a*;

- j) the type of sieve used;
- k) the drying time;
- l) the immersion period;
- m) the kind and temperature of water.

7 Verification

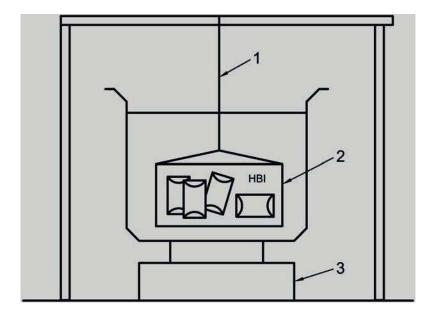
Regular checking of apparatus is essential to ensure test result reliability. The frequency of checking is a matter for each laboratory to determine.

The conditions of the following items shall be checked:

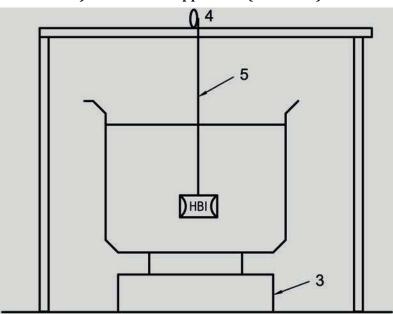
- sieves;
- weighing device;
- suspension device;
- thermometer.

It is recommended that internal reference material be prepared and used periodically to check test repeatability.

Appropriate records of verification activities shall be maintained.



a) Wire basket apparatus (method 1)



b) Wire suspension apparatus (method 2)

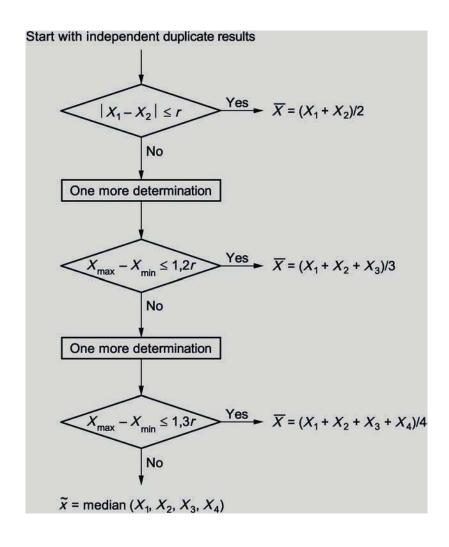
Key

- 1 suspension wire
- 2 wire basket
- 3 balance
- 4 wire tie post
- 5 thin wire

Figure 1 — Example of test apparatus (schematic diagram)

Annex A (normative)

Flowsheet of the procedure for the acceptance of test results



r: see <u>8.3</u>

